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Investigating the influence of ultrasound processing on drying kinetics and moisture migration measurement in lactobacillus cultured and uncultured beef jerky

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1 **Investigating the influence of ultrasound processing on drying kinetics and moisture**  
2 **migration measurement in lactobacillus cultured and uncultured beef jerky**

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16

## 17 Abstract

18 Low Frequency-Nuclear Magnetic Resonance (LF-NMR) was employed to elucidate changes in water  
19 distribution in cultured and uncultured beef jerky samples subjected to ultrasound pre-treatment..  
20 Ultrasound pre-treatment at frequencies of 25, 33 and 45 kHz for 30 min, followed by marination (18 h)  
21 was carried out for both uncultured and cultured (*Lactobacillus sakei*) jerky samples. Water mobility  
22 and distribution of water during drying were measured using LF-NMR. Among the various kinetic  
23 models assessed, the Wang and Singh model provided the closest fit to the drying experimental data,  
24 with high  $R^2$  ( $\geq 0.994$ ), low RMSE ( $\leq 0.023$ ) and low AICc ( $< -74.535$ ) values for both cultured and  
25 uncultured samples. Distributed exponential analysis of T2 transversal relaxation times measured by  
26 LF-NMR curves revealed the presence of three distinct peaks attributed to; bound water, water present  
27 within the dense myofibrillar protein matrix and free-water at a relaxation time range of 0– 10 ms (T2b),  
28 10– 100 ms (T21) and >100 ms (T22), respectively. Results presented in this study demonstrates that  
29 the ultrasound effect on drying behaviour was frequency dependent and that LF-NMR can be employed  
30 to evaluate moisture mobility and drying degree of beef jerky.

## 31 1. Introduction

32  
33 Beef jerky is a nutrient dense ready-to-eat meat snack, possessing characteristics of a typical  
34 intermediate moisture content product with a relatively long shelf-life. Commercially, beef jerky is  
35 prepared using a hurdle-technology approach which involves employment of interventions, such as;  
36 reducing water activity ( $a_w$ ) and addition of preservatives such as organic acids, spices and curing  
37 (nitrate/nitrite) salts. The development of whole-muscle and/or restructured jerky from a range of meats  
38 by employing various curing ingredients (e.g. as organic acids, spices, sugars, NaCl and nitrate/nitrite  
39 salts), curing methods and drying conditions have been widely reported (Choi, Jeong, Han, Choi, Kim,  
40 Lee, et al., 2008; Jang, Kim, Hwang, Song, Kim, Ham, et al., 2015; Kucerova, Hubackova, Rohlik, &  
41 Banout, 2015). Most recently, the application of starter culture (e.g. lactic acid bacteria) to improve

42 flavour and quality of jerky products, while preventing the growth of spoilage bacteria, has been  
43 reported (Biscola, Todorov, Capuano, Abriouel, Gálvez, & Franco, 2013; O'Connor, Ross, Hill, & Cotter,  
44 2015; Zhao, Zhao, Lu, Huang, He, Tan, et al., 2016).

45 The application of ultrasound has been reported to enhance mass transfer rates during brining/curing of  
46 meat, primarily by disrupting the continuity of cellular membranes due to various physical and chemical  
47 effects of ultrasound (C Ozuna, Cárcel, García-Pérez, Peña, & Mulet, 2015). Ultrasound, in combination  
48 with vacuum application has been shown to enhance the drying rate of beef and chicken meat (Başlar,  
49 Kılıçlı, Toker, Sağdıç, & Arici, 2014). Ultrasound pre-treatment is widely reported to accelerate drying of  
50 a range of food products (Awad, Moharram, Shaltout, Asker, & Youssef, 2012), which can affect texture  
51 and water activity of products. Additionally, ultrasound treatment has shown promise in improving meat  
52 tenderisation, depending on the ultrasonic intensities and processing times employed.

53         Moisture content is the main factor influencing the quality, safety and shelf life of meat-based  
54 jerky. Conventionally, the moisture content of commercial forms of jerky is determined by oven drying  
55 methods and sensory assessments. However, these methods are tedious, time-consuming, expensive  
56 and require trained and skilled personnel. Thus, there is a great scientific and industrial interest to  
57 develop a rapid, non-destructive and online method for determination of moisture content and drying  
58 degree in order to ensure consistent jerky quality. Low-field nuclear magnetic resonance (LF-NMR) is a  
59 sensitive, fast and non-invasive technique which has been widely adopted as an analytical technique  
60 for the characterization of water mobility and distribution within food matrices (Agudelo-Laverde et al.,  
61 2014; Troutman et al., 2001; Haiduc and van Duynhoven, 2005). The state and distribution of water in  
62 food matrices, including meat, can be determined by LF-NMR and can provide useful information  
63 about interactions between water and myofibrillar meat proteins, as it is governed by exchange of water  
64 protons and exchangeable protons in proteins (Bertram, Engelsen, Busk, Karlsson, & Andersen, 2004).  
65 LF-NMR has been successfully employed to study the effectiveness of various processing techniques,  
66 including; brining, cooking, freezing and thawing on water distribution and mobility (Bertram, Kohler,

67 Böcker, Ofstad, & Andersen, 2006; Damez & Clerjon, 2013; C. Li, Liu, Zhou, Xu, Qi, Shi, et al., 2012;  
68 Ojha, Keenan, Bright, Kerry, & Tiwari, 2016; Sánchez-Alonso, Moreno, & Careche, 2014). This  
69 technique has also been suggested as an alternative method for the conventional determination of  
70 drying degree upon the quality of chicken jerky (M. Li, Wang, Zhao, Qiao, Li, Sun, et al., 2014).

71 The objective of this study was to investigate the use of ultrasound as a pre-treatment prior to  
72 hot air convective drying of cultured and uncultured beef jerky. Modelling approaches were used to  
73 assess the influence of ultrasound frequency on the drying kinetics of beef jerky samples. Another  
74 objective of this study was to demonstrate a feasibility of using LF-NMR to determine water mobility and  
75 distribution of water during drying of cultured and uncultured beef jerky samples. Correlation analysis of  
76 transverse relaxation times and the moisture contents of dried beef jerky at different drying intervals  
77 were also determined to evaluate the drying degree of cultured and uncultured beef jerky samples.

78

## 79 **2. Materials and methods**

### 80 **2.1. Sample preparation and ultrasonic pre-treatment**

81 Beef used in this study was *Musculus Semitendinosus* which was obtained from a local supplier (Dublin  
82 Meat Company, Blanchardstown, Co. Dublin, Ireland). Meat was stored at 4°C, sliced to 0.2 cm in  
83 thickness using a meat slicer and were further cut by knife into slices of uniform dimensions (Length=  
84 10 cm, Width = 4 cm). The beef slices were cured using two different curing solutions: (I) Cultured,  
85 containing 70% water, *L. sakei* DSM 15831 culture, 1.5% salt, 1.0% sugar, 0.05% sodium nitrite and (II)  
86 Uncultured, containing 70% water, 1.5% salt, 1.0% sugar, 0.05% sodium nitrite (based on raw meat  
87 weight; v/w). The ingredients were thoroughly mixed, and samples from both cultured and uncultured  
88 treatment groups were subjected to ultrasonic (US) pre-treatments at frequencies of 25 kHz (Model:  
89 Elma IT H5), 33 kHz (Model: Jencons-PLS S1000) and 45 kHz (Model: Elma IT H5) for 30 min at  
90 comparable output power of circa 65 W along with a control (no US pre-treatment). US pre-treatments

91 were performed in ultrasonic bath systems maintained at a temperature of 30°C. All samples were  
92 subsequently cured for 18 h at 4°C.

93

## 94 2.2. Drying of Beef Jerky

95 Cultured and uncultured cured beef jerky slices were dried using a hot air drying oven (Gallendkamp  
96 Plus II, Weiss Technik, UK) at a temperature of 60°C for 4 h and using an air velocity which was  
97 maintained at 0.3 m/s. Beef jerky samples were placed in trays and were transferred to the hot air  
98 drying oven. Two slices from each treatment were withdrawn after every 30 min for 4 h and  
99 subsequently weight using precise weighing balance (Sartorius, Germany), after weight determination  
100 slices were placed back to the oven.

101

## 102 2.3. Mathematical modelling

103 Moisture content, on a dry basis, is the weight of moisture present in the product per unit weight of dry  
104 matter in the product. For drying experiments, where weight losses were recorded, the instantaneous  
105 moisture contents at any given time can be obtained from Eq.1:

$$106 \quad M = \frac{(M_o + 1)W_o}{W_t} - 1 \quad \text{Eq. 1}$$

107 Where  $W_o$  is the initial weight (g) of jerky sample after a curing period of 18 h,  $W_t$  is the weight (g) of  
108 sample at time  $t$  (min) and  $M_o$  is the initial moisture content (g water/g dry solids), respectively. The  
109 initial moisture content was determined using the hot air oven method as per AOAC. The data obtained  
110 experimentally for control and ultrasound pre-treated beef jerky slices from both uncultured and  
111 cultured groups were plotted as a dimensionless variable moisture ratio (MR) versus time as calculated  
112 from Eq. 2:

$$113 \quad \text{Moisture ratio (MR)} = \frac{(M_t - M_e)}{(M_o - M_e)} \quad \text{Eq.2}$$

114

115 Where  $M_t$  is the moisture content at any time  $t$ ,  $M_e$  the equilibrium moisture content and  $M_0$  is the initial  
 116 moisture content and all expressed as g water/g dry solids. The value of the equilibrium moisture  
 117 content ( $M_e$ ) is relatively small compared to  $M_t$  or  $M_0$ . Thus, Eq. (1) can be simplified as  $MR =$   
 118  $M_t/M_0$  (Ju, El-Mashad, Fang, Pan, Xiao, Liu, et al., 2016; Xie, Mujumdar, Fang, Wang, Dai, Du, et  
 119 al., 2017). Moisture diffusivity ( $D_f$ ) for beef jerky samples were calculated by using Eq. 3 by analogy to  
 120 the analytical solution to the Fick's second law of diffusion assuming negligible shrinkage, constant  
 121 temperature, and constant moisture diffusivity (Zielinska & Michalska, 2016) .

$$122 \quad MR = \frac{8}{\pi^2} \exp \left[ -\frac{\pi^2 D_f t}{4L^2} \right] \quad \text{Eq.3}$$

123

124 Where,  $D_f$  is the effective moisture diffusivity ( $m^2/\text{min}$ ),  $L$  is the thickness of the sliced beef (m).

125

126 Six empirical models were employed to describe drying kinetics were Henderson and Pabis, Wang and  
 127 Singh, Page, Lewis (Newton), Weibull and Peleg (Table 1). The regression coefficient ( $R^2$ ), Root mean  
 128 square error (RMSE) and AICc (Akaike information criterion) values were calculated using Eq. 4 – 6,  
 129 respectively.  $R^2$ , RMSE and AICc values were used as the primary criteria for measuring best model  
 130 fit.

$$R^2 = \frac{\sum_{i=1}^N (MR_i - MR_{pred,i}) \times \sum_{i=1}^N (MR_i - MR_{exp,i})}{\sqrt{[\sum_{i=1}^N (MR_i - MR_{pred,i})^2] \times [\sum_{i=1}^N (MR_i - MR_{exp,i})^2]}} \quad \text{Eq.4}$$

$$RMSE = \sqrt{\frac{1}{N} \sum_{i=1}^N (MR_{exp,i} - MR_{pred,i})^2} \quad \text{Eq.5}$$

$$131 \quad AICc = 2n - 2 \log_e (\mathcal{L}(\hat{\theta}|y)) + \frac{2n(n+1)}{N-n-1} \quad \text{Eq. 6}$$



132

133 Where,  $MR_{exp,i}$  is moisture content observed experimentally and  $MR_{pre,i}$  is predicted moisture  
134 content;  $SSE$  is the sum of squared error,  $2\log_e(\mathcal{L}(\hat{\theta}|y))$  is the log-likelihood at its maximum point of  
135 the model estimated,  $N$  and  $n$  represent the number of observations and parameters assessed,  
136 respectively.

## 137 2.2. LF-NMR transverse relaxation measurements

138 LF-NMR transverse relaxation measurements were carried out using a method described by  
139 McDonnell, Allen, Duggan, Arimi, Casey, Duane, et al. (2013) using a Maran Ultra instrument (Oxford  
140 Instruments, Abington, Oxfordshire, UK) resonating at a frequency of 23.2 MHz. Transverse relaxation  
141 ( $T_2$ ) times were measured using Carr-Purcell-Meiboom-Gill (CPMG) pulse sequence with the resultant  
142 relaxation decays analysed by tri-exponential unsupervised fitting using RI Win-DXP software  
143 (Version 1.2.3 Oxford Instrument, Abington, Oxfordshire, UK).

144

## 145 2.4. Statistical data analysis

146 Analysis of variance (ANOVA) was performed using SAS procedure (SAS Version 9.1.3, statistical  
147 Analysis Systems). Tukey's multiple comparison was used to compare treatment means. Pearson's  
148 correlation coefficients were analysed to determine a relationship between moisture content (MC, %) and  
149 TD-NMR relaxation parameters. Correlation coefficients and significance values were determined  
150 using PROC CORR (SAS Version 9.1.3).

151

## 152 3. Result and Discussion

### 153 3.1. Drying kinetics

154 The effects of ultrasound frequencies on drying kinetics of marinated (uncultured and cultured) beef  
155 jerky slices are shown in Figure 1(a) & 1(b), respectively. In general, the moisture ratio (MR) decreased

156 exponentially with time for control and ultrasound pre-treated samples from both cultured and  
157 uncultured groups. A variable effect was observed on the drying curves, depending upon culture  
158 treatment and ultrasonic frequency, as can be deduced from Figure 1. In general, a fast decrease in the  
159 MR [-] was observed for all treatments at initial stages followed by a slow decrease with drying time  
160 [min] at a drying temperature of 60°C. The moisture content decreased gradually for all samples, while  
161 a fast decrease in moisture content was observed at a frequency of 45 kHz, followed by the control, 25  
162 kHz and 33 kHz, respectively, for cultured samples. In the case of uncultured samples, control samples  
163 showed the fastest decrease in moisture content, followed by 45 kHz, 33 kHz and 25 kHz. Previous  
164 studies have shown that ultrasound pre-treatment can enhance drying rate for various food matrices  
165 (Fernandes, Rodrigues, García-Pérez, & Cárcel, 2015; García-Pérez, Cárcel, Benedito, & Mulet, 2007).  
166 However, the effect of ultrasound assisted drying depends largely on food matrix being dried, ultrasonic  
167 processing parameters and drying temperature. For example, ultrasound pre-treatment of various food  
168 matrices showed a significant decrease in drying time, whereas in some cases, minor improvements  
169 were reported (F. A. N. Fernandes, M. I. Gallão, & S. Rodrigues, 2008; A Mulet, Carcel, Sanjuan, &  
170 Bon, 2003). Generally, during the drying process, migration of moisture is fast due to the evaporation of  
171 surface moisture and decreases exponentially with an increase in drying time due to resistance offered  
172 by the matrix to moisture movement. In a study conducted by Başlar, Kılıçlı, Toker, Sağdıç, and Arici  
173 (2014), a significant decrease in drying time for ultrasound-assisted, vacuum-drying of chicken and  
174 beef meat samples was observed. There are several supporting studies which show that ultrasound  
175 enhances drying rate, owing to various mechanisms, thus modifying the diffusion boundary due to  
176 acoustic pressure waves, oscillating viscosities, compressions and expansions of materials leading to  
177 the formation of micro channels on surfaces which is required for fluid movement (Cárcel, García-  
178 Pérez, Benedito, & Mulet, 2012; A Mulet, Cárcel, Benedito, Rosselló, & Simal, 2003; Yao, 2016).  
179 Variation in drying rate in this study may be due to the diffusion of marination solution into the meat  
180 matrix due to the formation of micro channels on surfaces. Studies have shown that ultrasound

181 application can increase brine diffusion rate into a range of meat matrices (J. A. Cárcel, J. Benedito, J.  
182 Bon, & A. Mulet, 2007; A. Mulet, Cárcel, Sanjuán, & Bon, 2003; César Ozuna, Puig, García-Pérez,  
183 Mulet, & Cárcel, 2013). This may occur due to ultrasound assisted microinjection of brine into meat  
184 through the formation of microjets as a result of asymmetric cavitation near the solid surface of the  
185 product (Mason & Lorimer, 2002). However, it has been reported that no linear increase in diffusion of  
186 brine solution into meat matrices was observed with respect to ultrasonic intensity (McDonnell, Lyng,  
187 Arimi, & Allen, 2014).

188 The successful application of ultrasound on meat drying rates has been reported, however, the  
189 mechanism of action is not yet clear. In this study, the effect of ultrasound frequency on drying rate for  
190 both uncultured and cultured samples was probably due to the effect of ultrasound on lactobacillus  
191 culture and diffusion of marination solution into the beef jerky samples. A significant moisture change  
192 was observed in marinated beef jerky samples after 18 h marination for ultrasonic pre-treated samples  
193 compared to fresh beef (72.0%). For uncultured samples treated, at the lowest ultrasound frequency  
194 (25 kHz), a gain of 6.04% was observed whereas for 33 kHz and 45 kHz pre-treatments moisture gains  
195 of 5.60 % and 6.15%, respectively, were observed. In the case of cultured samples, no significant  
196 moisture gain was observed for the control group, whereas moisture gains of 5.12%, 4.11% and 3.58%  
197 were observed for ultrasound pre-treatments 33 kHz, 25 kHz and 45 kHz, respectively.

198 The observed changes were mainly due to uptake of marination solution. Similar gains in moisture have  
199 been reported for ultrasound pre-treatment prior to drying of fruit (F. A. Fernandes, M. I. Gallão, & S.  
200 Rodrigues, 2008; Oliveira, Gallão, Rodrigues, & Fernandes, 2011). However, in some cases, solid  
201 losses during ultrasound pre-treatments were also reported (Kadam, Tiwari, & O'Donnell, 2015;  
202 Oliveira, Gallão, Rodrigues, & Fernandes, 2011). A concentration gradient of soluble solids between  
203 beef slices and the marination solution resulted in water gain after pre-treatment and subsequent  
204 incubation. Increase in moisture uptake has been reported for marinated beef products, including; pork,  
205 poultry and beef, depending on composition of marination solution. Aktaş and Kaya (2001) observed an

206 increase in moisture uptake for beef *Longissimus dorsi* muscle after marination at 4°C for 24 h. In this  
207 study, moisture uptake was observed for ultrasound pre-treated samples, whereas no significant  
208 change in moisture uptake was observed for control samples. Research carried out by J. Cárcel, J.  
209 Benedito, J. Bon, and A. Mulet (2007) on ultrasound-assisted brine diffusion of pork muscle showed no  
210 significant change in moisture uptake in samples subjected to static brining and found that moisture  
211 uptake was dependent on ultrasonic intensity at a constant frequency of 20 kHz. Limited studies with  
212 muscle-based foods have, like this present study, also highlighted moisture uptake as a result of  
213 ultrasound pre-treatment in the case of Halal and non-Halal chicken breast (Leal-Ramos, Alarcon-Rojo,  
214 Mason, Paniwnyk, & Alarjah, 2011).

215

### 216 3.2. Drying models

217

218 Non-linear regression analysis was carried out for six drying models as a function of drying time and  
219 moisture ratio and various statistical parameters ( $R^2$ , RMSE and AICc) were determined to measure the  
220 goodness of model fit. Model and statistical parameters (of drying models are listed in Table 1. For all  
221 models  $R^2$  ranged from 0.941 to 0.998, RMSE ranged from 0.006 to 0.075 and AICc values ranged from  
222 -105.40 to -50.43. For beef jerky samples investigated, the Wang and Singh model had the closest fit  
223 to the drying experimental data, as evident from the high  $R^2$  values ( $\geq 0.994$ ) and the low RMSE ( $\leq$   
224 0.023) and low AICc ( $< -74.535$ ) values for both cultured and uncultured jerky samples. Model  
225 parameters ( $a$  and  $b$ ) obtained by fitting the Wang and Singh model indicated that the relative  
226 magnitude of the parameter accurately reflects drying behaviour. Drying constant values ( $a$ ) were in  
227 the range of  $-5.98 \times 10^{-3} \text{ min}^{-1}$  to  $-3.2 \times 10^{-3} \text{ min}^{-1}$  for uncultured and  $-6.73 \times 10^{-3} \text{ min}^{-1}$  to  $-3.39 \times 10^{-3}$   
228  $\text{min}^{-1}$  for cultured jerky samples, whereas, drying constant values ( $b$ ) varied from  $-4.22 \times 10^{-7} \text{ min}^{-2}$  to  
229  $9.28 \times 10^{-6} \text{ min}^{-2}$  for uncultured and  $1.23 \times 10^{-6} \text{ min}^{-2}$  to  $1.22 \times 10^{-5} \text{ min}^{-2}$  cultured samples. Model  
230 parameter ( $a$ ) was lowest in the case of 45 kHz and highest for 33 kHz for cultured samples, whereas,

231 in the case of uncultured samples it was lowest for control samples and highest for 25 kHz samples.  
232 The lower ( $a$ ) values reflect the higher moisture removal rates. A similar trend was also observed for  
233 drying kinetics when fitted to other models. Various models have been proposed to model drying  
234 kinetics of various food products, including; beef and chicken (Başlar, Kılıçlı, Toker, Sağdıç, & Arici,  
235 2014). Drying behaviour can be predicted using a range of models, however, in this study the Wang  
236 and Singh model was found to be the best fit. Best model fit can be judged based on various statistical  
237 parameters, however; AICc and RMSE values were the criteria used for model section, because  $R^2$   
238 alone cannot be judged for model fitting. AICc tends to have performance advantages over other  
239 criteria for model fitting (Burnham, Anderson, & Huyvaert, 2011). AICc value rise with an increase in the  
240 number of model parameters and the lower the AICc value, the better is the model performance. AICc  
241 criteria has been adopted by several researchers to test the performance of drying kinetics models  
242 (Buttchereit, Stamer, Junge, & Thaller, 2010; Gowen, Abu-Ghannam, Frias, & Oliveira, 2008; Kadam,  
243 Tiwari, & O'Donnell, 2015). The  $D_f$  value of the of cultured and uncultured beef samples ranged  
244 between  $0.90$  to  $1.33 \times 10^{-8} \text{ m}^2 \cdot \text{min}^{-1}$  and  $0.83$  to  $1.45 \times 10^{-8} \text{ m}^2 \cdot \text{min}^{-1}$ , respectively, as shown in Figure  
245 2. The highest  $D_f$  value was observed for control uncultured samples, and cultured samples pre-treated  
246 at 45 kHz.  $D_f$  value was found to increase with an increase in ultrasonic frequency in the case  
247 uncultured samples, however, values remained significantly lower for control jerky samples in all cases.  
248 Calculated  $D_f$  values were within the range ( $10^{-8}$  to  $10^{-10} \text{ m}^2/\text{s}$ ) of those previously reported for drying of  
249 biological materials (Başlar, Kılıçlı, Toker, Sağdıç, & Arici, 2014; Zogzas, Maroulis, & Marinou-Kouris,  
250 1996).

### 251 3.3. Water mobility by TD-NMR relaxometry

252 A representative LF-NMR  $T_2$  transverse measurement for uncultured and cultured samples after 18 h  
253 marination (i.e. before drying) and after the 4 h drying period is shown in Figure 3. Distributed  
254 exponential analysis of curve obtained for various samples revealed the presence of three distinct  
255 peaks obtained at relaxation time ranges of  $0$ – $10 \text{ ms}$  ( $T_{2b}$ ),  $10$ – $100 \text{ ms}$  ( $T_{21}$ ) and  $>100 \text{ ms}$  ( $T_{22}$ )

256 respectively. These peaks can be attributed to various fractions of water present in beef jerky samples.  
257 The first peak obtained at the shortest relaxation time ( $T_{2b}$ ) represents bound water which is closely  
258 associated with macromolecules (mainly proteins). The second peak at  $T_{21}$  represents water present  
259 within the dense myofibrillar protein matrix, whereas, the third peak at  $T_{22}$  can be attributed to free-  
260 water present outside the myofibrillar protein matrix. Presence of three water fractions at relaxation  
261 times and their association with muscle proteins has been previously reported (Huff-Lonergan &  
262 Lonergan, 2005; Pearce, Rosenvold, Andersen, & Hopkins, 2011). Ultrasound pre-treatment showed a  
263 shift in peaks for uncultured samples compared to cultured samples after 18 h of marination or 0 h  
264 drying (Figure 3a&b). In the case of cultured control samples, a higher level of bound water fraction was  
265 observed with a decrease in ultrasound pre-treated (Figure 3a), whereas, a shift in peaks were  
266 observed in the case of uncultured samples (Figure 3b). In this study, the largest fraction of water  
267 present in beef jerky samples was observed at  $T_{21}$  for cultured (in the range of 84.74–78.87%) and  
268 uncultured (90.51 to 66.47%) samples after 18 h of marination, whereas, during drying at 60°C, the  
269 proportion of water obtained at  $T_{21}$  was found to decrease with an increase in water proportion at  $T_{2b}$ .  
270 An increase in water fraction at  $T_{21}$  indicates an increase in the number of protons in the intra-  
271 myofibrillar space. Whereas, an increased water fraction at  $T_{22}$  population indicates a similar rise in  
272 number of protons, thereby representing an increase in the extra myofibrillar water population (Pearce,  
273 Rosenvold, Andersen, & Hopkins, 2011). An increase in the proportion of water at  $T_{2b}$  suggests a  
274 reduction in myofibrillar moisture and an increase in the bound water fraction obtained at  $T_{2b}$  due to the  
275 removal of myofibril and free-moisture during drying. Similar increases in the bound water fraction,  
276 indicating moisture mobility, was reported for beef granules during drying within a temperature range of  
277 40–60°C (X. Li, Ma, Tao, Kong, & Li, 2012). Analysis of variance showed that culture and drying time  
278 were the significant factors for all three relaxation times, whereas, ultrasound frequency was a  
279 significant factor for  $T_{21}$  ( $p=0.0001$ ),  $T_{22}$  ( $p=0.0010$ ) and an insignificant factor for  $T_{2b}$ . Interaction effects  
280 of drying time with culture and ultrasound frequency were significant for relaxation time and water

281 proportion. Similar, changes for water population at  $T_{21}$  and  $T_{22}$  relaxation times were also reported for  
282 ultrasound-assisted brining of pork samples in a study which concluded that a reduction in the  $T_{21}$   
283 population and an increase in the  $T_{22}$  population may be due to increased salt intake and a change in  
284 physical properties of meat during the curing process (Ojha, Keenan, Bright, Kerry, & Tiwari, 2016). The  
285 increased intake of curing solution owing to ultrasound pre-treatment can cause an enlarged  
286 electrostatic repulsion within myofibrils, thereby resulting in water mobility and osmotic dehydration  
287 (Vestergaard, Andersen, & Adler-Nissen, 2007).

288 A plot of moisture content (MC, %) and  $T_{22}$  relaxation time (free-water) indicated that a change in  
289 relaxation time is related to the MC of cultured and uncultured beef jerky samples (Figure 4). Similarly,  
290 (2014) showed a relationship between  $T_{21}$  and  $T_{22}$  with water holding capacity of tofu. Hence, moisture  
291 population data obtained from NMR can be used for indirect prediction of key moisture related  
292 measurements. In this study, a strong positive correlation was observed between MC and  $T_{22}$  ( $r=0.790$ ,  
293  $p<0.0001$ ) and proportion of water at  $T_{22}$  ( $P_2$ ) ( $r=0.709$ ,  $p<0.0001$ ) indicating that the MC of beef jerky  
294 samples is mainly associated with free-water. Correlation analysis also showed a strong positive  
295 relationship between drying time (h) and various water fractions and relaxation times (Table 2), with the  
296 exception of  $T_{2b}$ , whereas, a significant negative relationship was observed between water fraction  
297 associated with  $T_{2b}$ . This is probably due to a shift in relaxation time during the drying process.

#### 298 4. Conclusion

299 This study demonstrates that ultrasound pre-treatment have significant effect on drying behaviour and  
300 moisture mobility of cultured and uncultured beef jerky samples. However, improvement in drying rates  
301 for both cultured and uncultured samples was not evident from the drying models generated. Significant  
302 increases in moisture gain after ultrasonic pre-treatment promoted brine uptake due to the combined  
303 effect of cavitation and concentration gradient phenomena. Among several drying models tested to  
304 predict the drying behaviour of beef jerky samples, the Wang and Singh drying model was found to be

305 the best model as demonstrated by high  $R^2$ , low RMSE and AICc values. LF-NMR results showed  
306 moisture mobility during drying process with strong correlation with MC of jerky samples. LF-NMR can  
307 be employed to elucidate changes in water distribution and moisture content of beef jerky samples.

## 308 **Nomenclature**

309

310 LF-NMR: Low Frequency-Nuclear Magnetic Resonance

311  $W_0$ : Initial weight [g]

312  $W_t$ : Weight [g] at time t

313 t: time [min]

314  $M_0$ : Initial moisture content [g water/g dry solids]

315  $M_t$ : is the moisture content at any time t,

316  $M_e$ : Equilibrium moisture content

317  $D_f$ : Effective moisture diffusivity [ $m^2/min$ ],

318 L: The thickness of the sliced beef [m]

319 MR: Moisture ratio [-]

320  $R^2$ : The regression coefficient,

321 RMSE: Root mean square error

322 AICc: Akaike information criterion

323 MC: Moisture content [%]

324  $T_{2b}$   $T_{21}$  and  $T_{22}$ : Relaxation time (ms)

325

326

## 327 **5. References**



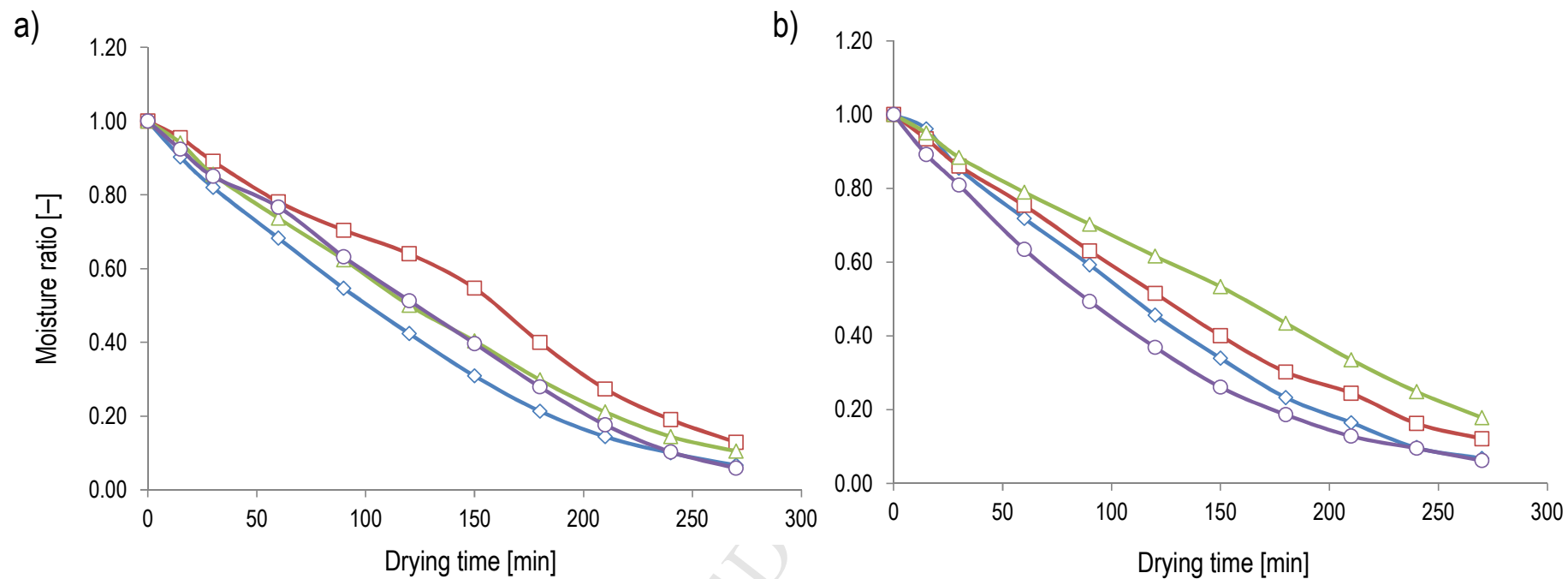
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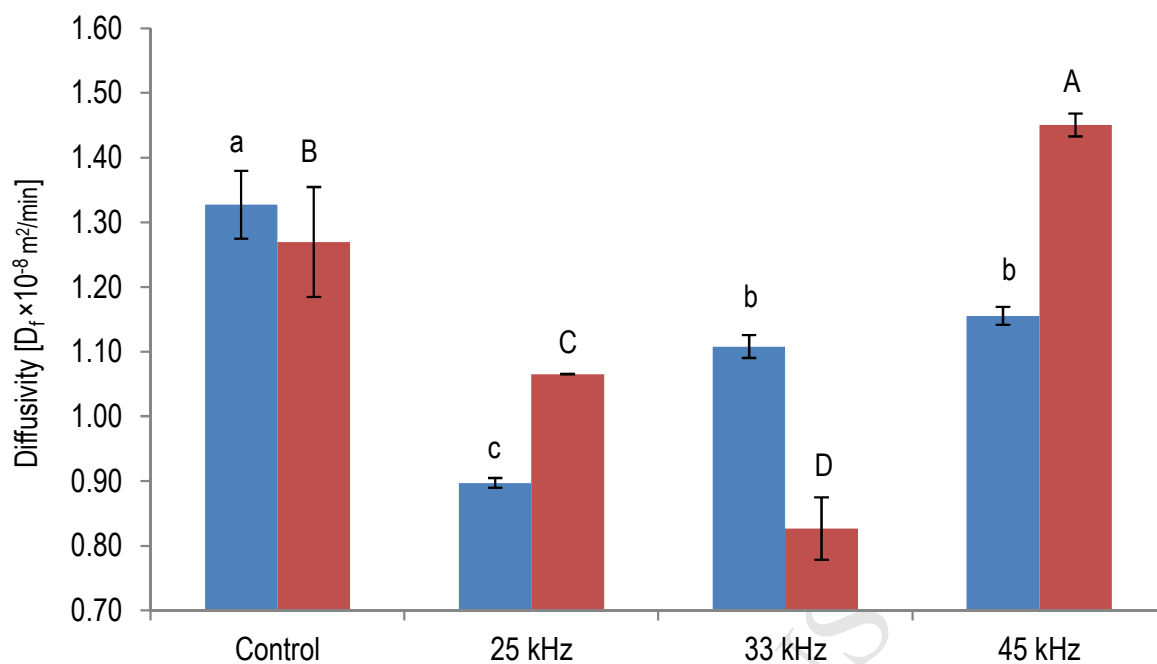
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461 Figure 1. Moisture ratio [MR] vs. drying time [min] for a) uncultured and (b) cultured beef jerky slices pre-treated at various ultrasonic frequencies [Control (◇), 25  
462 kHz (□), 33 kHz (△) and 45 kHz (○) respectively].



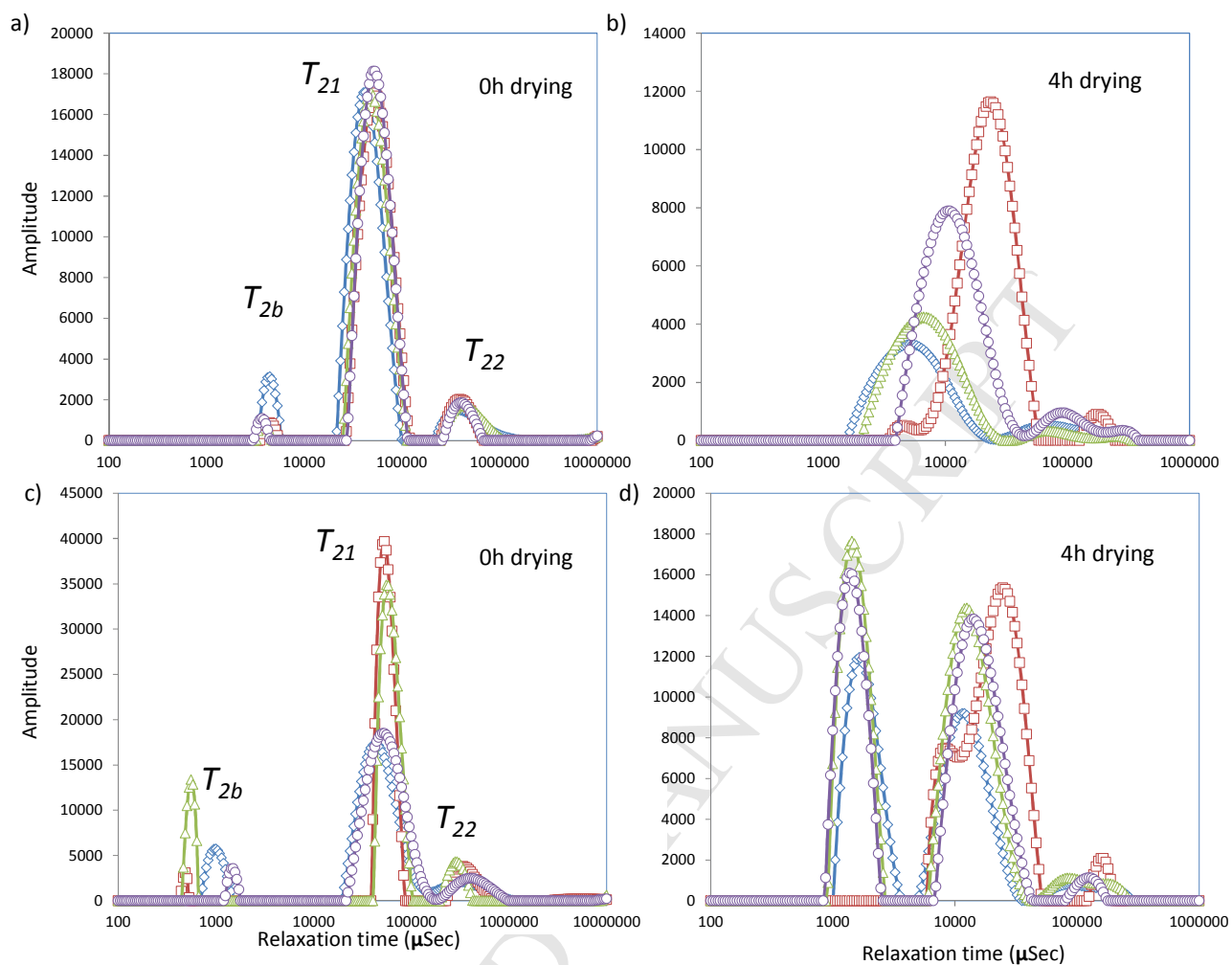
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464 Figure 2. Diffusivity for uncultured [■] and cultured [■] beef jerky samples pre-treated at various  
465 ultrasonic frequencies.

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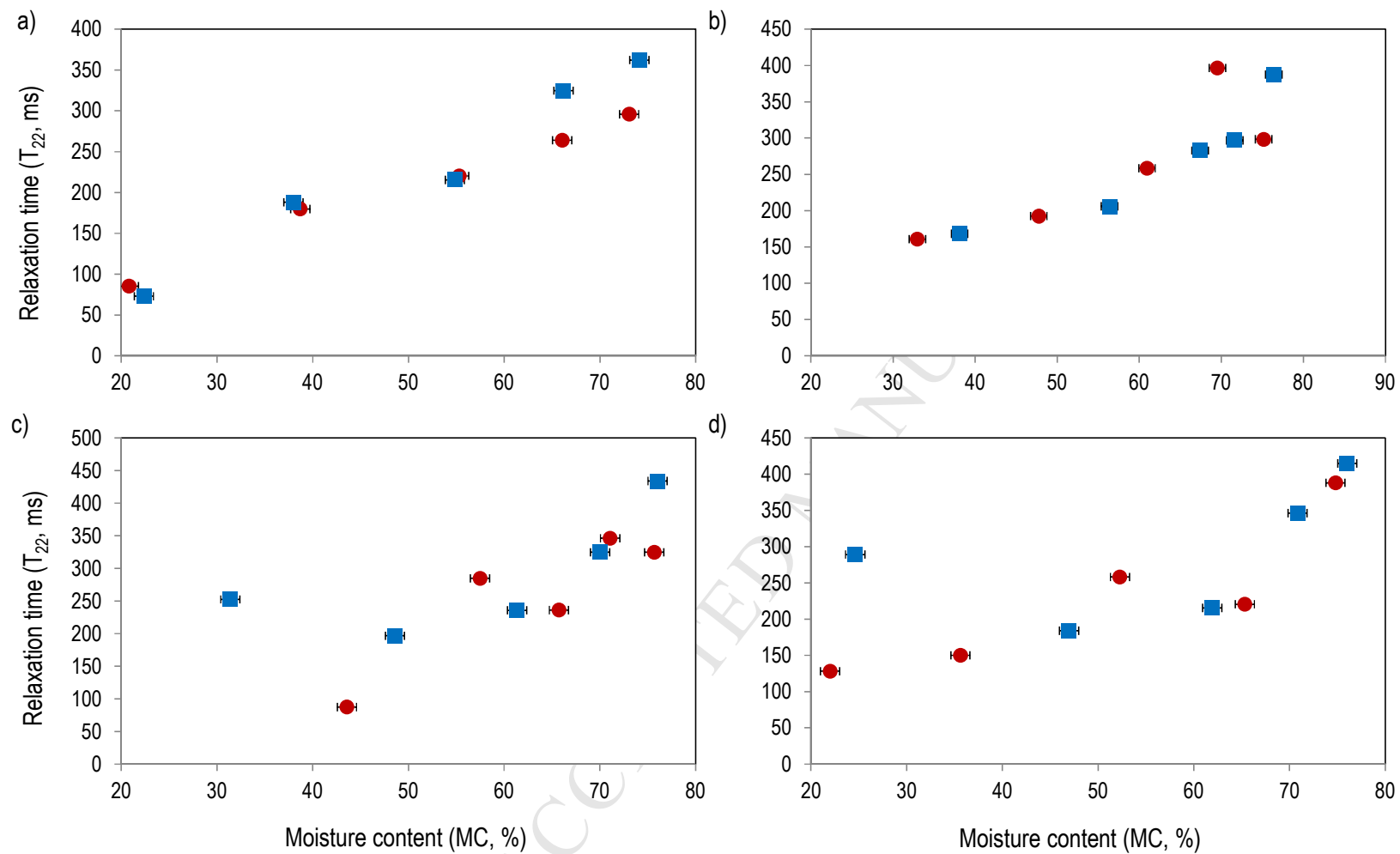
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470 Figure 3. Distribution of multi exponentially fitted transverse relaxation ( $T_2$ ) data for  
 471 uncultured (a – b) and cultured (c – d) beef jerky slices pre-treated at various ultrasonic  
 472 frequencies [Control ( $\diamond$ ), 25 kHz ( $\square$ ), 33 kHz ( $\Delta$ ) and 45 kHz ( $\circ$ ) respectively].

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474

475 Figure 4. Relationship between relaxation time ( $T_{22}$ ) and moisture content of beef samples during drying of cultured (●) and uncultured (●) control (a) and

476 ultrasound pre-treated beef jerky samples at 25 kHz (b), 33 kHz (c) and 45 kHz (d).

477 Table 1: Model parameters obtained from fitting drying models to beef jerky samples along with key statistical parameters

| Model                                     | Parameter   | Uncultured             |                       |                       |                        | Cultured               |                        |                        |                        |
|---|-------------|------------------------|-----------------------|-----------------------|------------------------|------------------------|------------------------|------------------------|------------------------|
|   |             | Control                | 25 kHz                | 33 kHz                | 45 kHz                 | Control                | 25 kHz                 | 33 kHz                 | 45 kHz                 |
| Henderson and Pabis<br>$MR = a \exp(-kt)$ | <i>a</i>    | 1.036                  | 1.057                 | 1.048                 | 1.056                  | 1.068                  | 1.045                  | 1.040                  | 1.031                  |
|   | <i>k</i>    | $8.18 \times 10^{-3}$  | $5.53 \times 10^{-3}$ | $6.83 \times 10^{-3}$ | $7.12 \times 10^{-3}$  | $7.82 \times 10^{-3}$  | $6.56 \times 10^{-3}$  | $5.09 \times 10^{-3}$  | $8.94 \times 10^{-3}$  |
|   | $R^2$       | 0.987                  | 0.950                 | 0.982                 | 0.963                  | 0.980                  | 0.984                  | 0.974                  | 0.994                  |
|   | <i>RMSE</i> | 0.038                  | 0.069                 | 0.043                 | 0.065                  | 0.049                  | 0.040                  | 0.046                  | 0.026                  |
|   | <i>AICc</i> | -64.230                | -50.435               | -60.915               | -51.810                | -58.080                | -62.630                | -59.260                | -71.965                |
| Wang and Singh<br>$MR = 1 + at + bt^2$    | <i>a</i>    | $-5.98 \times 10^{-3}$ | $-3.2 \times 10^{-3}$ | $-4.8 \times 10^{-3}$ | $-4.59 \times 10^{-3}$ | $-5.34 \times 10^{-3}$ | $-4.71 \times 10^{-3}$ | $-3.39 \times 10^{-3}$ | $-6.73 \times 10^{-3}$ |
|   | <i>b</i>    | $9.28 \times 10^{-6}$  | $-4.2 \times 10^{-7}$ | $5.28 \times 10^{-6}$ | $3.74 \times 10^{-6}$  | $6.71 \times 10^{-6}$  | $5.22 \times 10^{-6}$  | $1.23 \times 10^{-6}$  | $1.22 \times 10^{-5}$  |
|   | $R^2$       | 0.999                  | 0.994                 | 0.999                 | 0.996                  | 0.997                  | 0.999                  | 0.998                  | 1.000                  |
|   | <i>RMSE</i> | 0.010                  | 0.023                 | 0.011                 | 0.020                  | 0.019                  | 0.011                  | 0.012                  | 0.006                  |
|   | <i>AICc</i> | -95.105                | -74.535               | -90.375               | -78.980                | -79.380                | -90.780                | -89.385                | -105.400               |
| Page<br>$MR = \exp(-kt^n)$                | <i>k</i>    | $2.49 \times 10^{-3}$  | $3.17 \times 10^{-4}$ | $1.38 \times 10^{-3}$ | $6.56 \times 10^{-4}$  | $1.05 \times 10^{-3}$  | $1.38 \times 10^{-3}$  | $8.35 \times 10^{-4}$  | $3.76 \times 10^{-3}$  |
|   | <i>n</i>    | 1.250                  | 1.545                 | 1.319                 | 1.4785                 | 1.392                  | 1.3                    | 1.3465                 | 1.1725                 |
|   | $R^2$       | 0.997                  | 0.984                 | 0.997                 | 0.991                  | 0.998                  | 0.998                  | 0.991                  | 0.999                  |
|   | <i>RMSE</i> | 0.019                  | 0.039                 | 0.018                 | 0.032                  | 0.016                  | 0.016                  | 0.027                  | 0.010                  |
|   | <i>AICc</i> | -79.925                | -62.775               | -79.875               | -67.365                | -82.830                | -83.160                | -71.390                | -92.330                |
| Lewis (Newton)<br>$MR = \exp(-kt)$        | <i>k</i>    | $7.86 \times 10^{-3}$  | $5.13 \times 10^{-3}$ | $6.45 \times 10^{-3}$ | $6.68 \times 10^{-3}$  | $7.27 \times 10^{-3}$  | $6.22 \times 10^{-3}$  | $4.82 \times 10^{-3}$  | $8.64 \times 10^{-3}$  |
|   | $R^2$       | 0.984                  | 0.941                 | 0.977                 | 0.957                  | 0.972                  | 0.980                  | 0.969                  | 0.993                  |
|   | <i>RMSE</i> | 0.042                  | 0.075                 | 0.049                 | 0.071                  | 0.058                  | 0.045                  | 0.051                  | 0.029                  |
|   | <i>AICc</i> | -66.115                | -52.575               | -62.025               | -53.965                | -58.060                | -63.680                | -61.235                | -73.125                |
| Weibull<br>$MR = a \exp(-kt^n)$           | <i>a</i>    | 0.9737                 | 0.95545               | 0.9788                | 0.955                  | 0.9886                 | 0.9792                 | 0.97                   | 0.9864                 |
|   | <i>k</i>    | $1.81 \times 10^{-3}$  | $1.02 \times 10^{-4}$ | $9.56 \times 10^{-4}$ | $2.64 \times 10^{-4}$  | $8.83 \times 10^{-4}$  | $9.80 \times 10^{-4}$  | $4.47 \times 10^{-4}$  | $3.20 \times 10^{-3}$  |
|   | <i>n</i>    | 1.323                  | 1.7515                | 1.382                 | 1.6445                 | 1.425                  | 1.363                  | 1.463                  | 1.2025                 |
|   | $R^2$       | 0.998                  | 0.988                 | 0.997                 | 0.994                  | 0.998                  | 0.998                  | 0.993                  | 0.999                  |
|   | <i>RMSE</i> | 0.016                  | 0.034                 | 0.016                 | 0.026                  | 0.015                  | 0.013                  | 0.023                  | 0.009                  |
|   | <i>AICc</i> | -78.310                | -60.655               | -77.165               | -66.835                | -78.525                | -81.595                | -68.960                | -89.970                |
| Peleg<br>$MR = 1 - t/(a + bt)$            | <i>q</i>    | 149.2                  | 312.1                 | 199.65                | 214.1                  | 177.75                 | 203.05                 | 293.45                 | 122.55                 |
|   | <i>b</i>    | 0.48645                | -0.03865              | 0.34795               | 0.23575                | 0.37325                | 0.3582                 | 0.12383                | 0.58215                |
|   | $R^2$       | 0.997                  | 0.994                 | 0.998                 | 0.996                  | 0.995                  | 0.998                  | 0.998                  | 0.997                  |
|   | <i>RMSE</i> | 0.017                  | 0.023                 | 0.014                 | 0.021                  | 0.024                  | 0.014                  | 0.012                  | 0.017                  |
|   | <i>AICc</i> | -81.455                | -74.535               | -85.955               | -77.730                | -73.750                | -84.980                | -89.590                | -80.710                |



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479 Table 2. Correlation analysis showing a relationship between various parameters

|          | Time (h) | $P_0$    | $P_1$      | $P_2$      | $T_{2b}$             | $T_{21}$            | $T_{22}$             | MC (%)               |
|----------|----------|----------|------------|------------|----------------------|---------------------|----------------------|----------------------|
| Time (h) | 1.000    | 0.507*** | -0.437**   | -0.762**** | 0.206 <sup>ns</sup>  | -0.400*             | -0.822****           | -0.929****           |
| $P_0$    |          | 1.000    | -0.994**** | -0.468**   | 0.144 <sup>ns</sup>  | 0.282 <sup>ns</sup> | -0.305 <sup>ns</sup> | -0.615****           |
| $P_1$    |          |          | 1.000      | 0.366*     | -0.136 <sup>ns</sup> | -0.323*             | 0.249 <sup>ns</sup>  | 0.557****            |
| $P_2$    |          |          |            | 1.000      | -0.123               | 0.205 <sup>ns</sup> | 0.565**              | 0.709****            |
| $T_{2b}$ |          |          |            |            | 1.000                | 0.386 <sup>ns</sup> | 0.214 <sup>ns</sup>  | -0.205 <sup>ns</sup> |
| $T_{21}$ |          |          |            |            |                      | 1.000               | 0.702****            | 0.340*               |
| $T_{22}$ |          |          |            |            |                      |                     | 1.000                | 0.790****            |
| MC (%)   |          |          |            |            |                      |                     |                      | 1.000                |

480 ns:Not significant; \*P&lt;0.05; \*\*P&lt;0.01; \*\*\*P&lt;0.001; \*\*\*\*P&lt;0.001

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## Research Highlights

1. Drying behaviour is ultrasonic frequency dependent
2. Ultrasound can enhance marination rates
3. LF-NMR can be employed for water mobility and drying degree of beef jerky.